STABILITY OF THE DISPERSED BAND IN A SHALLOW LAYER SETTLER

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ABSTRACT

The present study is aimed at shallow-layer settlers, involving data collecting about the thickness of the dispersion band at different points along its length and its use in simulation work. At steady state, after step inputs in the agitation power, the length of the dispersed band varied in time, and didn't return to its initial condition. Although clean, pure fluids were used, *crud* formation was observed and acquired relevance especially for long period experiments. This behavior brought out the fact that *crud* formed during the experimentation disturbed the stability of the dispersed band. *Crud* analysis, performed by fluorescence microscopy, suggested the presence of bacteria. Thus, in order to make the experimental data reliable and reproducible, the effects of the formation and accumulation of *crud* at the water-organic interface must be taken into consideration, controlled and minimized by using appropriate biocides.

INTRODUCTION

Simulation of the hydrodynamics of mixer-settler units requires a mathematical representation of the dispersion behavior in the mixing and settling chambers and has great relevance for proper design and performance analysis. While mixing models and algorithms are well established, scarce work has been published regarding settler modeling [1-3], since application to particular physical-chemical systems requires accurate knowledge, obtainable only through detailed adequate experimentation and determination of the relevant parameters.

The analysis of the dynamic behavior of rectangular gravity settlers, where the dispersion flow is primarily horizontal, is complicated. Thus, because the drop velocity, the dispersion band thickness, and the drop size distributions vary along the length of the settler [4]. Mathematically, this corresponds to a formulation in terms of partial differential equations (distributed parameters model), contrasting with the simpler, concentrated parameters models used for the simulation of the compartmented extraction columns.

Usually, gravity settlers are designed for steady state operation using experimental data obtained from small-scale batch tests, without pilot-scale tests. However, transient conditions frequently occur in industrial continuous-flow systems, due to uncontrolled variations in feed-rate and/or in agitation speed; such transient regimes may imply variations in the thickness and length of the dispersion band, which, in turn, may have damaging consequences. Thus, it is important for the designer to understand the effects of such variations in order to achieve safe design and trouble-free operation.

Several observations focused on the study of *crud* influence at the industrial equipment performance have shown that it may modify some dispersion characteristics such as coalescence velocity which affect phases separation efficiency [5-6].

Some oscillations in the dispersion bands characteristics in settlers have been justified by formation of *crud* at aqueous organic interfaces in some industrial installations [5-7] and this fact modifies the drop coalescence. This *crud* layer composition is not well known and may be due to some solid particles and chemical and/or biological impurities. This *crud* layer may affect phase continuity, emulsion stability and air distribution during extraction. Ultimately it may initiate phase inversion, making correct work impossible [5]. So, *crud* presence increases organic phase losses, and so increases operation costs. Its formation is a complex process not yet well known.

However in pilot plant installations were pure products were used similar layers were also present [8]. In our work, various problems were found in experimentation, one of the more relevant being *crud* formation that was observed and acquired great importance especially for long period experiments. This behavior brought out the fact that crud formed during the experimentation disturbed the stability of the dispersed band.

EXPERIMENTAL PROCEDURE

The main initial objective of the present long term research program was to measure settler parameters, such as length and thickness of the dispersion band as well as drop diameter distribution, drop velocity along the settler and the response of these variables to disturbances in the operating conditions, in order to acquire parameter values for accurate modeling.

For this purpose, an acrylic pilot scale mixer-settler was purpose-designed and built, as reported in Baptista [8]. The installation work in closed circuit (fig 1), with the liquid-liquid system water-kerosene, that flow from two separate drums being pumping to an well agitated mixer where the kerosene is dispersed in the water, flowing together to the settler, where the two phases are separated then returning to the drums.

To determine the settler parameters a standard procedure was followed. The installation was in operation for several days (sometimes weeks) and during this process, a lot of *crud* was formed in the kerosene – water interface (at the end of the dispersion band, near discharge) which disturbed the settler performance (figs 2A , 2B). It was observed that the *crud* formation had an effect in order to diminish the dispersion band length. When that *crud* was removed the band grew up, but it was quite impossible to stabilize it. By this occurrence the main objective was postponed and the experimentation focus was changed to understand the *crud* nature and try its elimination, if possible.

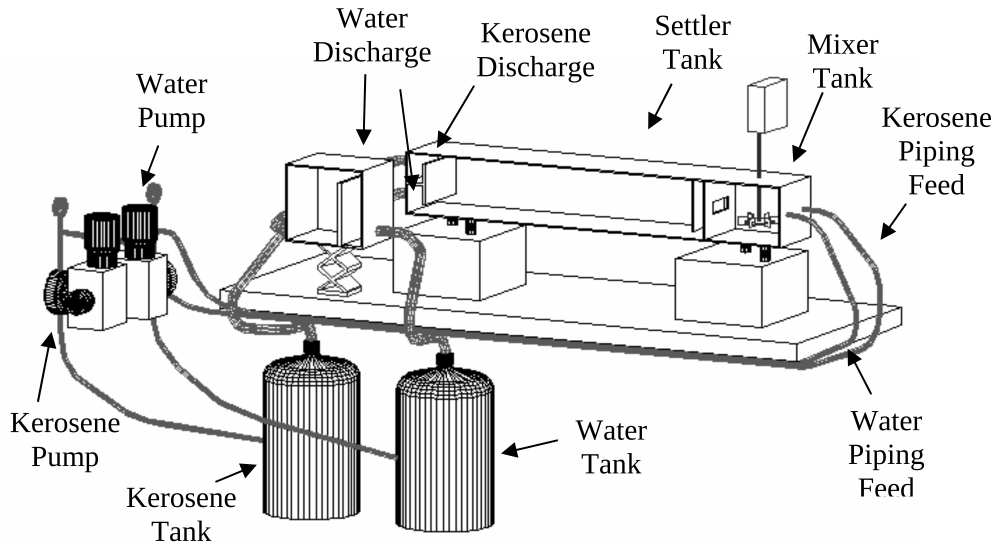

Figure 1. Experimental setup.

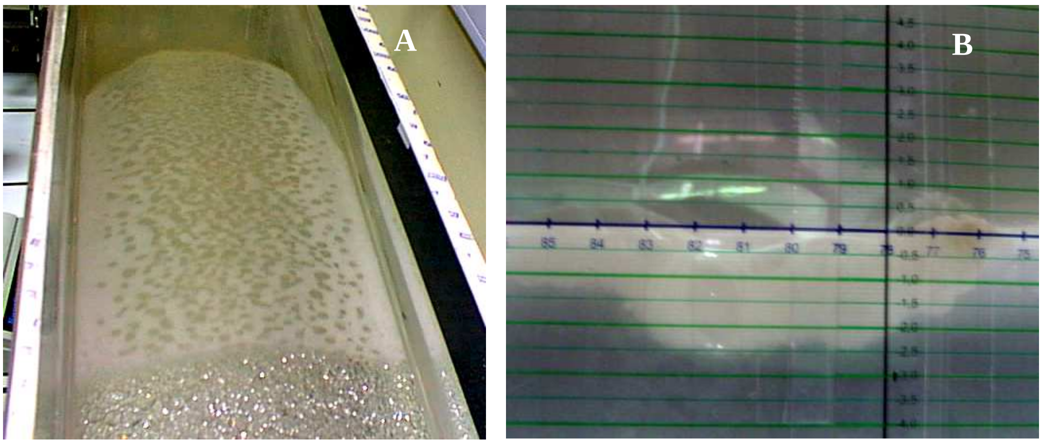

Figure 2. (A) Top view of the settler showing the accumulated *crud*, **(B)** Side view of the settler showing phases turbidity and *crud* accumulation.

The products used were clean (without solid particles) and pure (deionized water and commercial kerosene); however, with the time, a colorless and gelatinous-like *crud* was formed as described above. In order to investigate the possible presence of microorganisms, *crud* was heated at 65ºC, during one hour, and then 4.5 μmol l^{-1}

Propidium Iodide (PI) was added. PI binds to DNA, by intercalating between the bases, exhibiting an orange-red fluorescence [9].

After the biological nature of the *crud* has been identified, the next step was a first attempt to use a biocide in order to minimize its formation. Consequently, starting from extremely unfavorable system conditions (dirty phases and a lot of *crud* accumulated), the *crud* was mechanically removed and 4 drops of biocide were added. The biocide used was KURITA F-5106 (Enkrott), which is an aqueous soluble biocide. After this procedure the *crud* accumulation rate markedly diminished and the dispersion band length grew up. After more 260 hours (11 days) 12 additional drops of biocide were added. The experiment was carried on for 8 days more. This experiment results are shown in fig 5.

RESULTS AND DISCUSSION

The analysis of *crud*, by fluorescence microscopy, after treatment with PI, revealed the presence of orange, rod-like structures, which strongly suggests the presence of bacteria (Fig 4). This observation is in agreement with the description of growing of bacteria, yeasts and filamentous fungi in different fuels, namely in aviation kerosene [10]. Microorganisms might have entered in the system via the air, contaminated settler tank walls or being already present in the commercial kerosene used in the experiment. The growth of bacteria in the interface water-kerosene was most likely due to the presence of water, oxygen (introduced in the mixer, which provides enough oxygen dissolved in both phases) and carbon source (kerosene); nitrogen and phosphorous, could be present, as contaminants, allowing the microorganisms growth and thus the development of the gelatinous-like material (*crud)*.

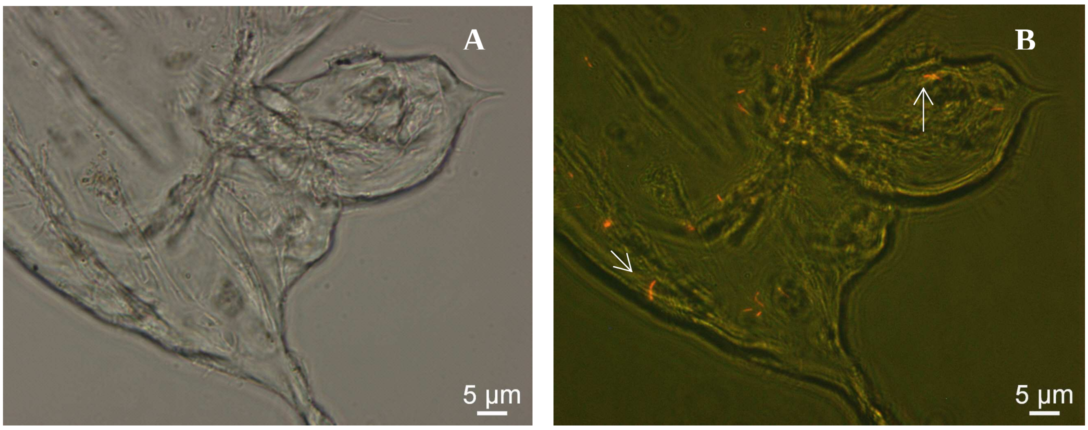

Figure 4. Photomicrographs of *crud*, treated with Propidium Iodide. Phase-contrast (A) and fluorescence plus phase-contrast (B) observations. Arrows: visualization of rod-like structures (bacteria) in the *crud*.

Given the impossibility of totally avoiding *crud* formation, the only experimental strategy that may be used is to control and prevent its growth.

Fig 5 shows the results of biocide utilization over *crud* development and its effect over the dispersion band length and stabilization under steady state conditions.

As said before the experiment was achieved using dirty phases with a lot of *crud*. Without *crud* removal the dispersion band length was not stabilized and its length was less than 76 cm. By removing the *crud* and adding biocide the length increased, the *crud* development rate was seen to diminish and the dispersion band attained a value between 85 and 88 cm do not being completely stabilized. A second addition of biocide, without *crud* removal, lead to a short instability followed by the complete stabilization of the dispersion band length at 84 cm. No significant further *crud* formation was observed. The shorter length after this second biocide addition may be justified by use of excess biocide and/or by an interfacial tension variation. However, many more systematized and well planned experiments are necessary to better understand this process.

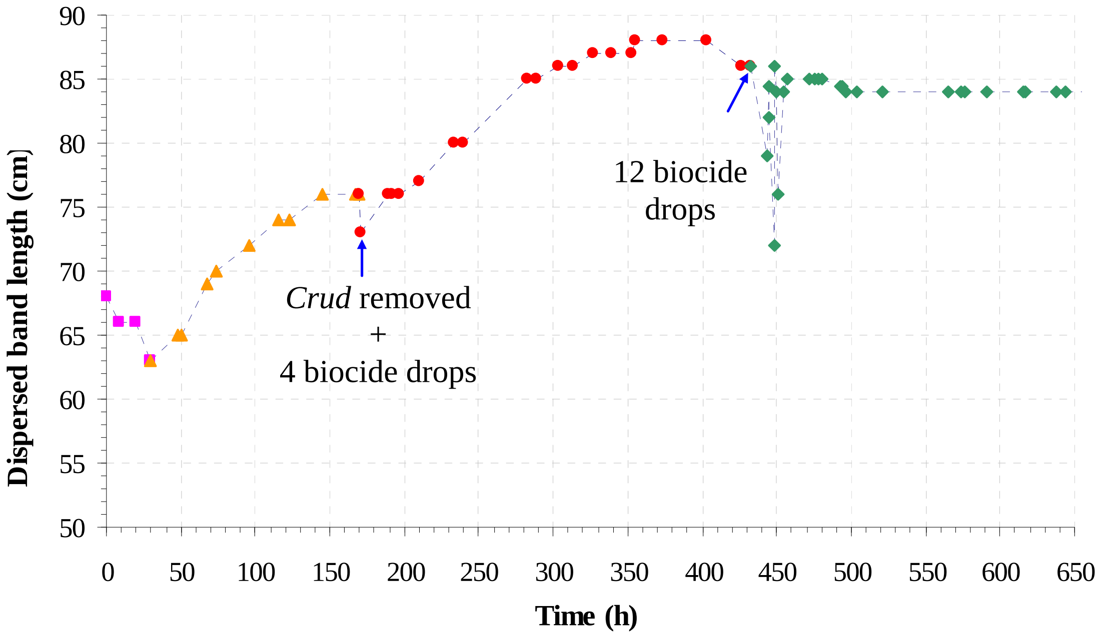

Figure 5. Behaviour of the settler dispersed band length as function of the time.

CONCLUSIONS

(1) Experiments using pure water and kerosene phases, in closed circuit and permanent operating conditions, have shown that band dispersion length stabilization was not attained, even at long experimentation times. *Crud* formation at the interface was observed. (2) The *crud* formation did not allow the normal progression of dispersion band and its removal, by means of invasive methods, leading to a permanent instability in the length of the dispersion band. (3) Fluorescent microscopy analysis suggests the

presence of bacteria in the gelatinous-like material (*crud*). (4) Preliminary experiments using a biocide revealed a dramatic decrease in *crud* formation and allowed the dispersion band stabilization. (5) Future work must be focused on the utilization of an adequate biocide so detailed experiments using different biocides must be carried out and its influence on dispersion properties (namely interfacial tension) analyzed.

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